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Dec 21, 1993

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TITLE: Field dependent fluids containing electrically conductive tin oxide coated materials

Brief Summary Text (60):

As set forth above, the inorganic substrates, can include a wide variety of materials. Further examples of inorganic oxides for use in the manufacture of catalysts are for example, cordierite, silicon carbide, silicon nitride, titania (such as anatase), alumina (preferably gamma alumina), titania and silica, magnesium aluminate spinel, mordenite, i.e., zeolite, silica, magnesia and mixtures thereof. The inorganic substrates, particularly the inorganic oxide supports are particularly useful and can be coated with a tin oxide forming component and converted to a tin oxide.

Brief Summary Text (69):

The tin oxide/substrate combinations, e.g., the tin oxide coated substrates, of the present invention are useful in other applications as well. Among these other applications are included porous membranes, resistance heating elements, electrostatic dissipation elements, electromagnetic interference shielding elements, protective coatings, field dependent fluids and the like.

Brief Summary Text (70):

One particularly useful feature of the present porous membranes is the ability to control the amount of tin oxide present to provide for enhanced performance in a specific application, e.g., a specific contacting process. For example, the thickness of the tin oxide-containing coating can be controlled to provide such enhanced performance. The coating process of the present invention is particularly advantageous in providing such controlled coating thickness. Also, the thickness of the tin oxide-containing coating can be varied, e.g., over different areas of the same porous membrane, such as an asymmetric porous membrane. In fact, the thickness of this coating can effect the size, e.g., diameter, of the pores. The size of the pores of the membrane or porous substrate may vary inversely with the thickness of the coating. The coating process of the present invention is particularly useful in providing this porosity control.

Brief Summary Text (78):

The porous membrane, in particular the substrate, can be predominately organic or inorganic, with an inorganic substrate being suitable for demanding process environments. The porous organic-containing membranes often include a porous organic based polymer matrix material having incorporated therein a three dimensional tin oxide-containing material, preferably including an electronically conductive tin dioxide coating, more preferably incorporating a dopant and/or a catalytic species in an amount that provides the desired function, particularly electrical conductivity, without substantially deleteriously affecting the properties of the organic polymer matrix material. These modified polymer membranes are particularly useful in porous membrane and/or electromembrane and/or catalytic processes.

Brief Summary Text (79):

Among the advantages of such membranes are: stability at high temperature and/or at

large pressure gradients, mechanical stability reduced and even substantially no compaction of the membrane under pressure), stability against microbiological attack, chemical stability especially with organic solvents, steam sterilization at high temperatures, backflush cleaning at pressures of up to 25 atm, and stability in corrosive and oxidation environment.

Brief Summary Text (80):

As noted above, selectivity can be enhanced by applying an electrical field onto the surface of the membrane. The electrical field is obtained using a two electrode electrical device; the conductive membrane constituting one of the two electrodes (anode or cathode--preferably anode). Such porous membranes can be obtained with one or more electronically conductive tin oxide-containing thin layers on a porous substrate. Conductive tin oxide combined with other metal oxide mixtures also provide improved properties for porous membranes and exhibit electronic conductivity, as well as other functions, such as catalysts or resistance heating.

Brief Summary Text (117):

The siloxanes or silicones useful as fluids are represented by the general structure ##STR7## wherein R.sub.14, R.sub.15, R.sub.16, R.sub.17, R.sub.18, R.sub.19, can each be alkyl, substituted alkyl, aryl and substituted aryl radicals and n' is a whole number from about 0 to about 2000 or more. Typical examples of alkyl and haloalkyl radicals are those heretofore described. Typical examples of the siloxanes are poly(methyl) siloxane, poly(methyl, phenyl) siloxane, poly(methyl, chlorophenyl) siloxane and poly(methyl,3,3,3-trifluoropropyl) siloxane.

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Jul 17, 1990

DOCUMENT-IDENTIFIER: US 4941893 A

**** See image for Reexamination Certificate ****TITLE: Gas separation by semi-permeable membranesAbstract Text (1):

A method is disclosed for separating gaseous silicon compounds from hydrogen and/or hydracids. Specifically, the method comprises the utilization of semi-permeable membranes for such gaseous separation. Particularly preferred is a composite membrane comprised of a coating separation layer of sulfonated polysulfone and a support layer of polysulfone. Mixtures of hydrogen and silane are particularly suitable for being separated by means of composite membrane separation.

Brief Summary Text (3):

This invention pertains to the field of gas separation and, more particularly, gas separation by the use of semi-permeable membranes. More specifically, the present invention relates to the separation of gaseous components which are frequently present in various gas stream mixtures found in the production and/or reaction of polycrystalline and/or epitaxial silicon metals.

Brief Summary Text (15):

More particularly, these gaseous components may be separated by contacting such a gaseous stream with a suitable semi-permeable membrane to effect such separation. Such a semi-permeable membrane may include an asymmetric membrane having a thin separation layer which determines the overall gas separation characteristics of the membrane. Alternatively, the semi-permeable membrane may also include a composite membrane comprised of a porous support layer having substantially no separation characteristics with respect to the gaseous components and a substantially non-porous separation layer positioned on the support layer which substantially determines the selective permeation characteristics of the overall composite membrane.

Brief Summary Text (21):

contained in said gaseous mixture which comprises contacting the gaseous mixture with one surface of a semi-permeable membrane which exhibits selective permeation of the at least first gas over that of the at least second gas, and removing from the vicinity of the opposite surface of the membrane a permeate having a concentration of the at least first gas which is greater than the concentration of the at least first gas in the gaseous mixture.

Brief Summary Text (22):

In preferred embodiments of the present invention, composite membranes are utilized to carry out the specified separation.

Brief Summary Text (23):

A particularly preferred composite membrane which provides excellent separation and permeation as well as excellent chemical stability and resistance to the gaseous silicon components and the hydracids is a separation layer comprised of sulfonated-polysulfone and a support layer comprised of polysulfone.

Drawing Description Text (2):

FIG. 1 is a schematic diagram of a polysilicon production process utilizing a membrane separator in accordance with the present invention to separate silane from hydrogen.

Drawing Description Text (3):

FIG. 2 is a schematic diagram of a process in which an intermediary stream containing a mixture of at least trichlorosilane and hydrogen from a silicone production process is treated so as to recover trichlorosilane utilizing a membrane separator in accordance with the present invention.

Drawing Description Text (4):

FIG. 3 is a schematic diagram of an apparatus used for measuring the permeability of a gas through a semi-permeable membrane.

Drawing Description Text (5):

FIG. 4 is a graph showing the permeability of trichlorosilane through a sulfonated polysulfone composite semi-permeable membrane as a function of time as measured by the apparatus of FIG. 3.

Drawing Description Text (6):

FIG. 5 is a graph showing the permeability of nitrogen and helium through a sulfonated polysulfone composite semi permeable membrane as a function of time as measured by the apparatus of FIG. 3, both before and after the membrane had been exposed to trichlorosilane.

Drawing Description Text (7):

FIG. 6 is a graph showing the permeability of hydrogen chloride through a sulfonated polysulfone composite semi-permeable membrane as well as the permeabilities of both nitrogen and helium, respectively, both before and after the membrane was contacted with the hydrogen chloride.

Drawing Description Text (8):

FIG. 7 is a graph showing hydrogen recovery as a function of stage cut through a sulfonated polysulfone composite semi-permeable membrane for a given set of feed conditions for a feed containing hydrogen, nitrogen and trichlorosilane.

Drawing Description Text (9):

FIG. 8 is a graph showing the concentration of nitrogen and trichlorosilane as a function of stage cut through the sulfonated polysulfone composite semi permeable membrane used for the measurements set forth in FIG. 5 for the same set of feed conditions.

Drawing Description Text (10):

FIG. 9 is a graph showing the concentration of hydrogen as a function of stage cut through the sulfonated polysulfone composite semi-permeable membrane used for the measurements set forth in FIG. 5 for the same set of feed conditions.

Drawing Description Text (11):

FIG. 10 is graph showing the size of the membrane surface area required for a given stage cut for the sulfonated polysulfone composite semi-permeable membrane used for the measurements set forth in FIG. 5 for the same set of feed conditions.

Detailed Description Text (4):

By virtue of the present invention, it has been discovered that semi-permeable membranes, particularly composite membranes, can effectively be utilized to separate these gaseous silicon compounds from the hydrogen or hydrogen chloride. As is well known to those skilled in the art of membrane technology, when referring to a number of gaseous components contained in a gas mixture, there may be a number of such components which will readily permeate through a semi permeable membrane at a rate which is much greater than other components contained in such gas mixture.

Those components which do, in fact, permeate at a relatively faster rate are typically referred to as "fast" gases for that particular gaseous mixture as compared to the "slow" gases which permeate at a lesser rate or not at all. With respect to a mixture of gaseous silicon compounds and hydrogen or hydrogen chloride, we have learned that the silicon compounds are "slow" gases as compared to the hydrogen and hydrogen chloride which are "fast" gases. As a result of appreciating that there is such a difference in permeation rates between the gaseous silicon compounds and the hydrogen or hydrogen chloride, we have realized that semi-permeable membrane separation was possible. However, and most importantly, we have also discovered that there are membranes, particularly composite membranes, which not only have excellent separation and permeability characteristics but which are, additionally, chemically resistant to these gaseous silicon compounds which typically have a corrosive nature.

Detailed Description Text (10):

To achieve selective separation, the semi-permeable membrane must exhibit less resistance to the permeation of one or more components than that of at least one other component contained within the gaseous mixture. Thus, selective separation can provide preferential depletion or concentration of one or more desired components in the mixture with respect to at least one other component and therefore provides a product having a different proportion of the one or more desired components to the at least one other component than that proportion in the mixture.

Detailed Description Text (11):

However, in order for membrane separation of one or more desired components to be commercially attractive, the membranes must not only be capable of withstanding the conditions to which they may be subjected during the separation operation, but also must provide an adequately selective separation of the one or more desired components, i.e., a high separation factor, as well as a sufficiently high flux, i.e., a high permeation rate, so that the use of such a separation procedure is carried out on an economically attractive basis.

Detailed Description Text (12):

With respect to reactive, gaseous silane-containing streams, alteration of the chemical structure of the membrane may occur, particularly if a polymer-based membrane is utilized. Such alteration may sometimes be acceptable if it does not lead to deterioration of long term membrane performance. By virtue of the present invention, as a preferred embodiment, it has been determined that composite membranes, particularly sulfonated polysulfone on polysulfone composite membranes, are capable of providing desirably high separation factors and high permeation rates with respect to the gaseous components discussed above, including excellent chemical stability.

Detailed Description Text (13):

Asymmetric type membranes are comprised essentially of a single permeable membrane material distinguished by the existence of two distinct morphological regions within the membrane structure. One region comprises a thin, dense semi permeable skin capable of selectively permeating one component of a fluid mixture. The other region comprises a less dense, porous, non selective support region that serves to preclude the collapse of the thin skin region of the membrane under pressure.

Detailed Description Text (14):

Composite membranes generally comprise a thin layer or coating of a suitable essentially non-porous membrane material superimposed on a porous substrate. This coating layer, also referred to herein as a separation layer, determines the separation characteristics of the composite structure, and is advantageously very thin so as to provide the desirably high permeability referred to above. The substrate or support layer only serves to provide a support for the membrane layer positioned thereon and has substantially no separation characteristics with respect

to the gaseous mixture being separated or concentrated.

Detailed Description Text (15):

These membranes may be fabricated in various shapes, such as (1) a flat sheet which may be supported in a typical plate and frame structure similar to a filter press; (2) a flat sheet rolled into spirals with spacing materials interleaved with the membrane and the assembly sealed to provide spiroidal channels permitting the passage of the feed on one side of the coiled membrane to the opposite side of the membrane; (3) as tubes lining the inner surface of a reinforced braid, the braid itself at times being a component in a larger tube; or (4) in the form of open-ended hollow fibers so organized and sealed into header plates so as to provide a separation of the flow over the external surfaces of the hollow fibers from any flow within the bores of the hollow fibers ensuing by virtue of passage of the gaseous feed mixture across the membrane. Such hollow fiber construction is preferred in the process of the present invention.

Detailed Description Text (16):

The invention is further described herein, for convenience of description, with particular reference to hollow fiber composite membranes. It will be understood, however, that the scope of the present invention is not limited to the use of the membranes in the composite structure in the hollow fiber form.

Detailed Description Text (17):

The hollow fiber membranes typically used in the art have continuous channels for fluid flow extending between the exterior and interior surfaces. Frequently, the pores have an average cross-sectional diameter less than about 20,000 Angstroms and in some hollow fibers the cross-sectional diameter is less than about 1,000 or 5,000 Angstroms. Advantageously, the walls of the hollow fibers are sufficiently thick that no special apparatus is required for their handling. Frequently, the hollow fibers may have outside diameters of about 20 to 1,000 microns, generally about 50 to 1,000 microns, and have walls of at least about 5 microns in thickness, generally about 50 to about 1,000 microns thick. The wall thickness in some hollow fibers may be up to about 200 or 300 microns. The coating may have a thickness ranging from about 0.01 to about 10 microns and preferably has a thickness of about 0.05 to about 2 microns.

Detailed Description Text (19):

The material used for the hollow fiber may be a solid, natural or synthetic substance. The selection of the material for the hollow fiber may be based on the heat resistance and/or mechanical strength of the hollow fiber, as well as other factors dictated by the separation process of the present invention and the operating conditions to which it will be subjected. Most importantly, the materials used, whether it be the porous support layer or the essentially non porous coating layer must be chemically resistant to each of the gaseous silicon compounds and hydracids noted above. The hollow fibers may be flexible or substantially rigid.

Detailed Description Text (22):

The polysulfone or other hollow fiber substrates employed in the practice of particular embodiments of the present invention can be prepared in accordance with conventional techniques well known in the art. Hollow fibers are generally spun from a dope composition of the desired fiber polymer, quenched, washed and dried. As disclosed by Cabasso, et al. in "Composite Hollow Fiber Membranes", Journal Of Applied Polymer Science, Volume 23, 1509-1525 (1979), and in "Research and Development of NS-1 and Related Polysulfone Hollow Fibers for Reverse Osmosis Desalination of Seawater", Gulf south Research Institute, July 1985, Distributed by National Technical Information Service, U.S. Department of Commerce Publication PB 248,666, polysulfone hollow fibers can be spun from a ternary solution of polysulfone, poly(vinyl pyrrolidone) and dimethylacetamide, with the total polymeric concentration in the solution desirably being 40 to 52 weight %, and the polysulfone/poly(vinyl pyrrolidone) ratio being 1.5:2.0. The well known tube in-

tube jet technique is disclosed as being suitable for the spinning procedure, with water at about 21.degree. C. being the preferred outside quench medium for the fibers. The quench medium in the center of the fiber is desirably air. Quenching is typically followed by washing the fibers, for example, conveniently with hot water at about 50.degree. to 60.degree. C. Following such washing, the hollow fibers are dried prior to being coated with the separation film layer to form the desired composite membrane. For this purpose, the polysulfone hollow fibers are typically dried by passage through a hot air drying column for a suitable period of time.

Detailed Description Text (23):

Hollow fiber substrates are typically substantially porous and the extent of their surface and bulk porosity is controlled by dry/wet, wet, dry or melt extrusion techniques which are well known to those skilled in the semi-permeable membrane art. The porosity of the hollow fibers may be further modified by solvent annealing or high temperature annealing techniques.

Detailed Description Text (24):

The coating layer of the composite membrane is in the form of an essentially non interrupted membrane, i.e., an essentially non-porous membrane, in contact with the porous support layer.

Detailed Description Text (25):

The materials for the coating may be natural or synthetic substances, and are often polymers. Synthetic substances include both addition and condensation polymers. Typical of the useful materials which can comprise the coating are polymers which can be substituted or unsubstituted, and which are solid or liquid under gas separation conditions, and include synthetic rubbers; natural rubbers; relatively high molecular weight and/or high boiling liquids; organic prepolymers; poly(siloxanes) (silicone polymers); polysilazanes; acrylonitrile-containing copolymers; polyesters (including polyarylates); cellulosic polymers; polysulfones, especially modified polysulfones; poly(alkylene glycols) such as poly(ethylene glycol), poly(propylene glycol), etc.; polymers from monomers having α -olefinic unsaturation such as poly(olefins), e.g., poly(ethylene), poly(butadiene), poly(2,3-dichlorobutadiene), poly(isoprene), poly(chloroprene), poly(styrene)s including poly(styrene) copolymers, e.g., styrene butadiene copolymer, poly(vinyl halides) (e.g., poly(vinyl bromide)), poly(vinylidene halides), fluorinated ethylene copolymer, poly(arylene oxides), e.g., poly(xylylene oxide); polycarbonates; and any interpolymers including block interpolymers containing repeating units from the above, and grafts and blends containing any of the foregoing. The polymers may or may not be polymerized after application to the porous support layer.

Detailed Description Text (26):

Particularly useful materials for coatings comprise cellulose acetate, silicon rubber, and ethyl cellulose. Most preferably, a sulfonated polysulfone is utilized as the coating material for the composite membrane. Such sulfonated polysulfones are discussed in, for example, U.S. Pat. No. 3,709,841, U.S. Pat. No. 4,054,707, U.S. Pat. No. 4,207,182, European Patent Application 0,202,849, European Patent Application 0,165,077 and European Patent Application 0,202,841 all of which are incorporated herein by reference as if set out in full. Sulfonated-polysulfones are also discussed in the Journal of Applied Polymer Science, Volume 20, pages 1885-1903 (1976) in an article entitled Sulfonated Polysulfone by A. Noshay, et al., the contents of which is also incorporated herein by reference.

Detailed Description Text (28):

Sulfonated bisphenol A polysulfone is particularly preferred as the coating for the separation layer for the composite membrane.

Detailed Description Text (33):

Sulfonated polyarylethersulfones with degrees of substitution between about 1.0 to

about 2.5 meq/g of dry polymer that are soluble in solvents such as methoxyethanol, nitromethane, and alcohol/water mixtures are particularly useful for the preparation of the composite membranes capable of effectively separating gaseous silicon compounds from hydrogen or hydrides.

Detailed Description Text (34):

The dried polysulfone hollow fiber is coated with the coating solution of the sulfonated-polysulfone and is then dried. Such a coating and drying sequence may comprise the technique used and described in the Coplan, et al. patent, U.S. Pat. No. 4,467,001, which is incorporated herein by reference. Thus, the dried hollow fibers are passed through the coating solution contained in a coating vessel, and is then passed through a drier oven and a cure oven for contact with drying air or other suitable gas, and higher temperature curing air or other gas prior to being taken up on a winder or otherwise being processed or stored for eventual incorporation in membrane modules suitable for use in the desired separation operation. For the coating of polysulfone hollow fibers with the sulfonated polysulfone, which is a preferred embodiment of the present invention, it is generally desirable to employ drying temperatures of from about 20.degree. C. to about 100.degree. C. Those skilled in the art will appreciate that it is also possible to dry the separation layer on the support layer without employing the separate curing step noted above.

Detailed Description Text (35):

In a preferred embodiment of the present invention, the support layer is subjected to a high temperature annealing process. Although it is preferable to anneal the substrate prior to its being coated with the separation layer, the annealing process may be carried out on the coated substrate as well. The resulting composite membrane formed from such an annealed substrate, provides for an even greater enhancement in both separation and permeation characteristics.

Detailed Description Text (37):

In use, the composite membrane will generally be assembled as part of a membrane separating device. The membrane device is designed to carry out a selective separation of at least one component from a fluid stream mixture. The membrane apparatus will typically consist of an enclosure and a membrane assembly positioned therein. The membrane assembly can be constructed in the form of a spiral wound cartridge, a hollow fiber bundle, a pleated flat sheet membrane assembly, and like assemblies common in the membrane industry. The membrane assembly is constructed so as to have a feed surface side and an opposite permeate exit side. The enclosure is constructed so as to enable the feed stream mixture to be brought into contact with the membrane feed-surface side. Conduit means are provided for the removal of the part of the feed stream that did not permeate through the membrane, and for the separate removal of the permeate components that have passed through the membrane.

Detailed Description Text (38):

In conducting the gas separations, including concentrations, of the present invention, the exit side of the membrane is maintained at a lower thermodynamic potential for the at least one permeating, fast component, i.e., H₂ or HX, than the thermodynamic potential at the feed side. The driving force for the desired permeation through the membrane is a differential in thermodynamic potential across the membrane, for instance, as provided by a differential in partial pressure. Permeating components pass into and through the membrane and can be removed from the vicinity of the exit side of the membrane to maintain the desired driving force for the permeation. The functionality of the membrane does not depend upon the direction of feed flow or the surface of the membrane which is first contacted by the gaseous feed mixture.

Detailed Description Text (39):

The gaseous mixture sent to the membrane separator can range from atmospheric to at least 2000 psig, generally about 50 to about 3000 psig, and preferably about 100 to

about 200 psig.

Detailed Description Text (42):

It is understood, of course, that in addition to the at least one hydrogen and/or hydracid gas that may be present in the gaseous mixture and the at least one gaseous silicon compound that is also present in the gaseous mixture, other fast and slow gases may be present as well. Accordingly, during the separation process utilizing the composite membrane, these extraneous fast gases may also be permeated through the permeation membrane in conjunction with the hydrogen and/or hydracid gases.

Detailed Description Text (43):

As used herein, it will be understood that the selectivity, or separation factor, of a membrane or membrane module assembly represents the ratio of the permeate rate of the more permeable (the fast gas) component to the less permeable (the slow gas) component of the gaseous mixture being separated which permeability is expressed in ft.sup.3 (STP)/ft.sup.2 .multidot. day .multidot. psi.

Detailed Description Text (44):

Typically, the permeation rate of hydrogen through the composite membrane at room temperature (25.degree. C.) may be anywhere in the range of from about 0.1 to about 10.0 ft.sup.3 (STP)/ft.sup.2 .multidot. day .multidot. psi, and more typically is in the range of from about 0.5 to about 4.0 ft.sup.3 (STP)/ft.sup.2 .multidot. day .multidot. psi. Of course, this permeation rate is dependent upon the process conditions, and most importantly is dependent upon the particular semi-permeable membrane being utilized.

Detailed Description Text (45):

Similarly, the permeation rate of hydracids such as hydrogen chloride at room temperature (25.degree. C.) is in the range of from about 0.1 to about 10.0 ft.sup.3 (STP)/ft.sup.2 .multidot. day .multidot. psi, and more typically in the range of from about 0.2 to about 5.0 ft.sup.3 (STP)/ft.sup.2 day .multidot. psi, and is again dependent upon the process conditions and the specific composite membrane being utilized.

Detailed Description Text (46):

Correspondingly, the separation factor between the fast gas components of the present invention, i.e., the hydrogen and hydracids, and the gaseous silicon compounds, is typically in the range of from about 20 to about 2,000, and more typically is in the range of from about 50 to about 500 (at 25.degree. C.), again dependent upon specific process conditions and the particular composite membrane being utilized.

Detailed Description Text (49):

In order to reduce the build up of hydrogen in the recycle loop, however, a purge stream 18 must generally be provided. In the prior art such purge stream would generally be flared and any silane contained therein would simple be lost. In the present invention, however, such loss is avoided by passing the purge stream containing both silane and hydrogen into a semi-permeable membrane separator 120.

Detailed Description Text (50):

Separator 120 is provided with a cellulose acetate composite membrane having a polysulfone substrate. The permeate, which is that material passing through the membrane, has a much higher concentration of hydrogen, which is a fast gas, as compared to the raffinate, which is that material which does not pass through the membrane and contains a high concentration of the silane, the slow gas in this feed system. The permeate, containing substantially hydrogen, is passed out of the separator via line 20. The raffinate, containing substantially silane, is passed out of the separator via line 22. The recovered silane in line 22 may be recycled back to reactor 100 (not shown) or utilized in any desired manner.

Detailed Description Text (53):

In the present invention, however, a membrane separator is utilized to accomplish a major separation of the trichlorosilane from the hydrogen to thereby produce a stream concentrated in trichlorosilane which is only then subjected to a refrigeration step. Clearly, the refrigeration unit in the process of the present invention is substantially smaller than that required by the prior art.

Detailed Description Text (54):

Accordingly, an intermediary stream containing trichlorosilane and hydrogen is first fed to a flash tank condenser 300 via line 30 to condense a portion of the trichlorosilane which leaves the condenser via line 32. The trichlorosilane/hydrogen stream, now containing a reduced amount of trichlorosilane, leaves the condenser via line 34, passes through heater 310 and is then introduced to membrane separator 320 via line 36. The stream is heated in heater 310 so as to prevent any condensation of trichlorosilane in the membrane separator. Condensation might occur if the partial pressure of the trichlorosilane in the raffinate is equal to the vapor pressure of the trichlorosilane. Membrane separator 320 contains a sulfonated polysulfone composite membrane.

Detailed Description Text (55):

The majority of the hydrogen contained in the gas mixture permeates through the membrane and leaves as the permeate through line 38. The raffinate now containing a concentrated amount of trichlorosilane with some hydrogen is then passed via line 40 into a pre-cooler 330 in preparation for being introduced into refrigeration unit 340 via line 42. In refrigeration unit 340, the trichlorosilane is separated from the remaining hydrogen by condensation. Substantially pure trichlorosilane leaves the refrigeration unit via line 44 and substantially pure hydrogen leaves the unit via line 46. The trichlorosilane is then processed in accordance with conventional techniques to form the desired silicone compositions.

Detailed Description Text (60):

To test the chemical stability of various membrane materials to the presence of silane, a particularly corrosive gaseous silicon compound, a number of membrane materials are exposed to silane in test cells for a period of time and then compared to untreated examples using the following techniques:

Detailed Description Text (69):

The apparatus is comprised of two sections, namely, a permeation assembly shown in FIG. 3 by dotted line 100 and a feed assembly designated by solid line 200. A bomb 210 contains liquid trichlorosilane. The permeation assembly includes a membrane separator 220 comprised of a sulfonated polysulfone coating on a polysulfone substrate composite membrane, a coil 230, and a pressure transducer 240. Valve 260 controls flow of material into the membrane separator. Valve 280 controls the flow of trichlorosilane leaving bomb 210, and valve 300 controls the flow of material entering through line 12.

Detailed Description Text (70):

Both the feed and permeation assemblies were placed in oven 500. The permeation assembly was wrapped with heating tape to keep its temperature $T_{sub.2}$ higher than the oven temperature $T_{sub.1}$ to prevent any condensation in the membrane separator. Checking for condensation was done through the pressure transducer. Thus, if, for example, $T_{sub.2}$ is kept at 55.degree. C. and $T_{sub.1}$ is set 50.degree. C., then if the pressure transducer shows the vapor pressure of trichlorosilane at 50.degree. C., then it is known that there is no condensation taking place in the membrane separator.

Detailed Description Text (74):

$P_{sub.0}$ = pressure on permeate side of membrane

Detailed Description Text (75):

P.sub.1 =pressure on feed side of membrane at time equal to zero, i.e., at the start of the experiment

Detailed Description Text (77):

At the start of the experiment, the ratio of $(P - P_{\text{sub.0}}) / (P_{\text{sub.1}} - P_{\text{sub.0}})$ is equal to 1. As the experiment continues, if the ratio becomes less than 1, this is an indication that the material being tested is permeable through the membrane. For a material that is not very permeable, the ratio of $(P - P_{\text{sub.0}}) / (P_{\text{sub.1}} - P_{\text{sub.0}})$ remains substantially close to 1.

Detailed Description Text (78):

For the trichlorosilane, as can be seen from FIG. 4, the results of the measurements show that it is impermeable inasmuch as the membrane pressure does not decrease with time. Instead, the membrane pressure increases slightly with time and then levels off. This slight increase in pressure is due to the permeation of nitrogen from the permeate side of the membrane. The nitrogen in the permeate side is used to purge moisture out of the membrane separator.

Detailed Description Text (80):

Once again using the apparatus of Example II, and further to the measurements taken in Example III, before and after the membrane separator was exposed to trichlorosilane, permeability measurements were made for both helium and nitrogen. The results of those measurements are shown in FIG. 5 using the same set of coordinates as FIG. 4.

Detailed Description Text (81):

The "I" and "II" set forth to the right of the graph in connection with He, and the "I", "II", and "III" set forth in connection with N.sub.2, respectively, refer to separate runs that were made with these materials through the membrane. The "I" run was considered a purging run.

Detailed Description Text (82):

As can be seen, after the membrane separator was contacted with the trichlorosilane, the helium permeability did not change at all while nitrogen permeability increased. However, after the membrane separator was repeatedly purged with nitrogen, the nitrogen permeability decreased with time and gradually approached the original permeability that was measured before the membrane was exposed to the trichlorosilane.

Detailed Description Text (84):

Once again using the permeating apparatus of Example II, the permeabilities of helium and nitrogen were measured, both before and after the membrane was exposed to hydrogen chloride. Hydrogen chloride permeability was also measured.

Detailed Description Text (86):

The results of these measurements reveal that hydrogen chloride permeates faster than helium and that the separation factor of hydrogen chloride with respect to helium is 1.2. Secondly, these results also show that the membrane separator is stable in the presence of hydrogen chloride inasmuch as the permeabilities of both the helium and the nitrogen remained constant, even after the membrane was exposed to the hydrogen chloride.

Detailed Description Text (88):

Hydrogen permeability and silane permeability through a 5 ft composite membrane comprising a cellulose acetate coating on a polysulfone substrate was also tested. The results are shown in Table I below.

Detailed Description Text (89):

As can be seen, the value for the separation factor of hydrogen with respect to

silane is 117. The silane permeability remains the same even after the membrane separator was exposed to silane for over 216 hours. This indicates that chemical interaction between the silane and the components of the composite membrane was not present and the membrane remains chemically stable.

Detailed Description Text (90):

The membrane separator was tested by using two hydrogen/silane gas mixtures: one containing 48.8 mole percent silane and the other containing 1.0 mole percent silane. The operating conditions of these two tests are set forth in Table II below.

Detailed Description Text (93):

Having determined the permeabilities for both hydrogen and trichlorosilane, a membrane separator can now be sized and the separation performance predicted.

Detailed Description Text (94):

The operating conditions of the membrane separator, using a sulfonated polysulfone coated polysulfone composite membrane with hollow fiber length of about 1 foot, are set forth in Table V below:

Detailed Description Text (95):

Because the trichlorosilane is not permeable through the membrane as determined earlier, it can be assumed that the separation factor of hydrogen with respect to trichlorosilane is about 2,000. Graphs showing the separation performance and the size of separator are shown in FIG. 7 through 10 which have been briefly described earlier.

Detailed Description Text (97):

FIG. 7 shows that 80% of the hydrogen can be removed from the feed at a stage cut equal to 0.58, i.e., the pinch point. The concentration of trichlorosilane, the concentration of nitrogen (FIG. 8), and the concentration of hydrogen in the permeate at the pinch point (FIG. 9) are 0.25%, 0.95% and 98.8%, respectively. Accordingly, from FIG. 10, it is determined that the surface area required for the membrane at the pinch point for the feed conditions noted in Table V is approximately 7,000 ft.².

Detailed Description Paragraph Table (2):

TABLE II		Operating Conditions for SiH.sub.4	
--H.sub.2 Membrane Separation Test 1 Test 2			
Feed Composition	48.8% SiH.sub.4 1% SiH.sub.4 (molar)	51.2% H.sub.2	99% H.sub.2
Feed Pressure	89.7 psia 89.7 psia	Permeate Pressure	25.7 psia 25.7 psia
34.about.37.degree. C.		34.about.37.degree. C.	

CLAIMS:

1. A method for separating at least a first gas selected from the group consisting of H.sub.2, HX and mixtures thereof contained in a gaseous mixture from at least a second gas selected from the group consisting of SiX.sub.a H.sub.b and mixtures thereof, where X = a halogen ion,

a = 0 to 4,

b = 0 to 4,

a + b = 4

contained in said gaseous mixture which comprises contacting the gaseous mixture with one surface of a semi-permeable membrane which exhibits selective permeation of the at least first gas over that of the at least second gas, and removing from

the vicinity of the opposite surface of the semi-permeable membrane a permeate having a concentration of the at least first gas which is greater than the concentration of the at least first gas in the gaseous mixture.

2. The method of claim 1, wherein the semi-permeable membrane is an asymmetric membrane or a composite membrane.

3. The method of claim 2, wherein the composite membrane comprises a porous support layer having substantially no separation characteristics with respect to the gaseous mixture and a substantially non-porous separation layer positioned on the support layer which substantially determines the selective permeation characteristics of the composite membrane.

14. The method of claim 2, wherein the asymmetric membrane comprises at least one of sulfonated-polysulfone, cellulosic polymers, cellulose acetate, synthetic and natural rubbers, polysiloxanes, polysilazanes, polyesters, polycarbonates, and mixtures thereof.

15. The method of claim 2, wherein the asymmetric membrane is a sulfonated polysulfone.

17. The method of claim 1, wherein the composite membrane is in hollow fiber form.

22. A method of separating hydrogen from silane contained in a gaseous mixture which comprises contacting the gaseous mixture with one surface of a semi-permeable membrane which exhibits selective permeation for the hydrogen over that of the silane and removing from the vicinity of the opposite surface of the semi permeable membrane a permeate having a concentration of hydrogen which is greater than the hydrogen concentration in the gaseous feed mixture.

23. The method of claim 22, wherein the semi-permeable membrane is an asymmetric membrane or a composite membrane.

24. The method of claim 23, wherein the composite membrane comprises a porous support layer having substantially no separation characteristics with respect to the gaseous mixture and a substantially non-porous separation layer positioned on the support layer to substantially determine the selective permeation characteristics of the composite membrane.

27. A method of separating hydrogen from trichlorosilane contained in a gaseous mixture which comprises contacting the gaseous mixture with one surface of a semi-permeable membrane which exhibits selective permeation for the hydrogen over that of the trichlorosilane and removing from the vicinity of the opposite surface of the semi-permeable membrane a permeate having a concentration of hydrogen which is greater than the hydrogen concentration in the gaseous feed mixture.

28. The method of claim 27, wherein the semi-permeable membrane is an asymmetric membrane or a composite membrane.

29. The method of claim 28, wherein the composite membrane comprises a porous support layer having substantially no separation characteristics with respect to the gaseous mixture and a substantially non-porous separation layer positioned on the support layer to substantially determine the selective permeation characteristics of the composite membrane.

32. A process for producing a semiconductor silicon metal from a chlorosilane selected from the group consisting of trichlorosilane, dichlorosilane and mixtures thereof, which comprises:

(a) providing the chlorosilane in admixture with hydrogen gas in a reaction zone

having a temperature sufficient to effect reduction and decomposition of said chlorosilane to silicon metal on a deposition surface; and then

(b) contacting exhaust gases leaving the reaction zone comprised of at least unreacted chlorosilanes, hydrogen, and by product hydrogen chloride with a semi-permeable membrane separator which exhibits selective permeation of the hydrogen and hydrogen chloride over the chlorosilanes, to produce a permeate having a concentration of hydrogen and hydrogen chloride which is greater than the concentration of hydrogen and hydrogen chloride in the exhaust gases, and a raffinate having a concentration of chlorosilanes which is greater than the concentration of such chlorosilanes in the exhaust gases.

34. The process of claim 32, wherein the exhaust gases are cooled prior to contacting the membrane separator to a temperature such that a portion of the trichlorosilane is condensed and removed.

37. The process of claim 32, wherein the semi-permeable membrane is an asymmetric membrane or a composite membrane.

38. The process of claim 37, wherein the composite membrane comprises a porous support layer having substantially no separation characteristics with respect to the gaseous mixture and a substantially non-porous separation layer positioned on the support layer which substantially determines the selective permeation characteristics of the composite membrane.

49. The process of claim 37, wherein the asymmetric membrane comprises at least one of sulfonated-polysulfone, cellulosic polymers, cellulose acetate, synthetic and natural rubbers, polysiloxanes, polysilazanes, polyesters, polycarbonates, and mixtures thereof.

50. The process of claim 37, wherein the asymmetric membrane is a sulfonated polysulfone.

52. The process of claim 32, wherein the semi-permeable membrane is in hollow fiber form.

55. A process for producing a semiconductor silicon metal from silane which comprises:

(a) providing the silane in admixture with hydrogen gas in a reaction zone having a temperature sufficient to effect reduction and decomposition of said silane to silicon metal on a deposition surface; and then

(b) contacting exhaust gases leaving the reaction zone comprised of at least unreacted silane and hydrogen with a semi-permeable membrane separator which exhibits selective permeation of the hydrogen over the silane to produce a permeate having a concentration of hydrogen which is greater than the concentration of hydrogen in the exhaust gases and a raffinate having a concentration of silane which is greater than the concentration of such silane in the exhaust gases.

57. The process of claim 55, wherein the semi-permeable membrane is an asymmetric membrane or a composite membrane.

58. The process of claim 57, wherein the composite membrane comprises a porous support layer having substantially no separation characteristics with respect to the gaseous mixture and a substantially non-porous separation layer positioned on the support layer which substantially determines the selective permeation characteristics of the composite membrane.

69. The process of claim 57, wherein the asymmetric membrane comprises at least one

of sulfonated-polysulfone, cellulosic polymers, cellulose acetate, synthetic and natural rubbers, polysiloxanes, polysilazanes, polyesters, polycarbonates, and mixtures thereof.

70. The process of claim 57, wherein the asymmetric membrane is a sulfonated polysulfone.

72. The process of claim 55, wherein the semi-permeable membrane is in hollow fiber form.

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END OF SEARCH HISTORY